TECHNICAL REPORT R-91

THE EFFECT OF REPEATED STRESSING ON THE BEHAVIOR OF LITHIUM FLUORIDE CRYSTALS

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SUMMARY

This paper is concerned with the mechanism of fatigue in crystals, and a brief review of present knowledge on this subject is given. The fact that results of reversed-bend tests on lithium fluoride single crystals at 2 cpm are similar to those previously obtained at 1,800 cpm indicates that speed effects do not play an important role in determining the behavior of lithium fluoride under reversed stressing. Reversed-torsion tests yield additional evidence that vacancy formation is not a primary cause of fatigue failure. Lithium fluoride is a material which does not readily cross slip. The absence of fatigue failures in both reversed bending and reversed torsion lends further support to the theory that cross slip is necessary in order to obtain usual fatigue behavior. The results further demonstrate that dislocation pileups do not lead to fatigue failure in lithium fluoride. The results also indicate that plastic deformation in the course of reversed cycling will relax an initially applied mean load. Etch-pit studies of lithium fluoride yield visual evidence of the growth of slipbands during cycling.

INTRODUCTION

GENERAL REVIEW OF FATIGUE MECHANISMS

Ever since the phenomenon known as fatigue of metals was recognized over a century ago, a search has been underway for the basic mechanism involved. Review of the research conducted during this period reveals the substantial progress that has been made and indicates that an understanding of the complex processes involved in fatigue is close at hand.

According to Cazaud (ref. 1), the earliest work on fatigue was done in 1829 by Albert who investigated the behavior of mine cable subjected to repeated stressing. Later work by Fairbairn and Wohler during the period from 1850 to 1860 brought out the concept of an endurance limit and showed that the number of cycles to failure depended upon the stress level above the endurance limit.

The first significant work pertaining to the mechanism involved in fatigue was done by Ewing and Humfrey (ref. 2) who found in 1903 that slip occurred during the fatigue process. The work of Gough (ref. 3) and his coworkers during the 1920's established the fact that the slip systems which are operative under cyclic loading are the same as those which are operative under unidirectional loading. Gough also found that the cracks which lead to final failure originate in the slipbands.

In 1939, Orowan (ref. 4) presented a theory of fatigue which involved a process of cumulative strain hardening whereby the local stress in critical regions could be increased until fracture in the region occurred. Based on the information available at the time, the theory offered a reasonable explanation of the observations. However, the research work of the 1950's has brought forth additional information which is not in support of the Orowan mechanism.

For example, Wood and Segall (ref. 5) found that when copper specimens were subjected to

[1] This paper is based in part upon a thesis entitled "An Investigation of the Mechanism of Fatigue in Crystals" submitted by Arthur J. McEvily, Jr., in partial fulfillment of the requirements for the degree of Doctor of Engineering Science, in the School of Engineering, Columbia University, May 1959.

repeated cycles of constant plastic strain amplitude, the yield stress was not raised indefinitely, as would be expected if the Orowan mechanism were operating, but instead leveled off at a value which was a function of plastic strain. Ebner and Backofen (ref. 6) obtained similar results for single crystals of copper not only at room temperature but also at 77° K.

Another objection to the Orowan theory is that it does not emphasize the role of the surface in fatigue. That the surface is indeed critical is evidenced by the beneficial effects of lifetime derived by intermittently interrupting a fatigue test to remove the surface layers. A further indication is the industrial practice of shot-peening to increase fatigue life. In addition, none of the methods of inspecting the interior of fatigue specimens, such as sectioning (ref. 7), small-angle X-ray scattering (ref. 8), and direct observation of transparent crystals (ref. 9), has ever resulted in the detection of voids or cracks entirely beneath the surface.

In view of these objections to the Orowan theory, there existed a need for a new formulation of a theory of fatigue which would take into account the recent observations. The key to the later theories was provided by Forsyth (ref. 10), who in 1953 found that material was extruded from the slipbands during the fatigue process. In further work he found that the intrusions also developed during the cyclic process. It was postulated that these effects would lead to the formation of stress raisers on the surface which would facilitate local fracture and growth of fatigue cracks.

Since the initial observations were made on age-hardening materials, it was thought that a process of overaging, leading to softening, was involved in the extrusion-intrusion process. However, additional work showed that the extrusion-intrusion process also occurred in nonaging materials. Of particular importance in assessing the role of thermal effects is the work of McCammon and Rosenberg (ref. 11), who found that fatigue occurred at temperatures as low as 4.2° K, and thereby clarified the theoretical picture by eliminating thermally activated processes such as dislocation climb, vacancy diffusion, and oxidation as necessary to the fatigue process, although such processes could play a modifying role at higher temperatures. For example, Wilson and Forsyth (ref. 12) have recently found evidence of a recovery process operating during the cyclic loading of pure aluminum.

With respect to the influence of temperature on the basic mechanism, the work of Hull (ref. 13) with copper crystals indicates that essentially the same processes are involved at room temperature as are involved at 4.2° K. He examined the surface structure of slipbands at 293°, 90°, 20°, and 4.2° K and found that the shape and size of extrusions did not appear to be affected by the temperature at which the specimens were fatigued.

The extrusions and intrusions found by Hull consisted of thin ribbons or crevices about 0.05μ thick. Their height or depth varied widely, but the maximum value was about 1.5μ. Since these effects were observed at 1 percent of the estimated life, Hull considered that the major portion of the fatigue lifetime was spent in the propagation of fatigue cracks. However, McEvily and Machlin (ref. 14) have pointed out that for much of the lifetime the growth is crystallographic in nature. They consider that such crystallographic cracks are simply large intrusions which grow by the same mechanism which caused their formation. In this view there is no real distinction between crystallographic crack initiation and growth since both are manifestations of the same process. Only in the final stages when noncrystallographic cracks are formed does crack propagation by some localized fracturing mechanism operate.

With respect to the morphology of extrusions and intrusions, Forsyth (ref. 9), using silver chloride, found that extrusions and intrusions usually formed on the same plane and near together. Recently, Wadsworth and Hutchings (ref. 15), using copper, found that extrusions were more common than intrusions, some of the extrusions apparently having formed in regions isolated from other intrusions or extrusions. Less often, extrusions and intrusions occurred alternately along the same slipband. Wadsworth and Hutchings also obtained experimental evidence that the slipbands described by Thompson, Wadsworth, and Louat (ref. 16), which persisted after the surface layers of cyclically loaded specimens were polished away, may have been shallow cracks which resulted from the intrusion process.

Another factor of importance in the formation of extrusions and intrusions is the relationship of the slip vector to the surface. Experiments by Wadsworth with cadmium (see Discussion in ref. 17) and by Ebner and Backofen with copper (ref. 6) have shown, for cases in which the slip direction of the primary slip system was in the plane of the surface, that the extrusion-intrusion process does not occur in that system but instead occurs in some other system whose slip direction does not lie in the plane of the surface.

The results of such tests indicate that the process of extrusion-intrusion formation is a fairly widespread phenomenon which plays an important role in the fatigue mechanism. However, it may not be the only process. For example, in the fatigue of pure aluminum small cylindrical cavities extending from the surface within slipbands have been observed. These cavities are quite different in appearance from intrusions and may indicate that a different mechanism is operative. In addition, Wood (ref. 18) considers the fatigue mechanism to be a function of stress level, with extrusion-intrusion the important process at low stress levels and something akin to the Orowan mechanism of cumulative strain hardening operative at high stress levels. Whether there are two such mechanisms, or whether the extrusions and intrusions formed at high stress levels are merely difficult to observe because of the superimposed effects of large plastic deformation has not yet been clearly established.

In recent years two dislocation models have been developed to explain the formation of extrusions and intrusions during cyclic loading, one by Cottrell and Hull (ref. 19) and the other by Mott (ref. 20). These models incorporate the principal aspects of the recent experimental findings and are considered to be important in understanding the fatigue mechanism.

In the Cottrell-Hull model, extrusion and intrusion formation is a cooperative process involving the sequential motion of edge dislocations on intersecting slip planes. The motion of the dislocations is such that as material moves in along one slip plane to form an intrusion, material simultaneously moves out along another slip plane to form an extrusion. In this model, therefore, no buildup of material or void formation occurs within the specimen.

In the Mott mechanism a screw dislocation which intersects the surface traverses a closed circuit in the course of a fatigue cycle. The motion around such a circuit requires that cross slip of the screw dislocation take place. Depending upon the nature of the circuit, extrusions or intrusions can be formed, but in the simplest version of the model this would involve void formation or a buildup of material within the specimen. Later variations of the model (ref. 14) overcame this difficulty by linking extrusion formation to intrusion formation.

In order to determine the relative validity of these two models, McEvily and Machlin (ref. 14) tested two groups of single crystals. All crystals were oriented to satisfy the conditions of the Cottrell-Hull mechanism, but one group of crystals was characterized by an ability to cross slip easily whereas the second group was not. The results of the fatigue tests showed that in the first group extrusions and intrusions were found and fatigue failures occurred, whereas in the second group no extrusions or intrusions were found and fatigue failures did not occur. These results, therefore, indicated that cross slip is an important requirement for a fatigue mechanism.

AIMS OF PRESENT INVESTIGATION

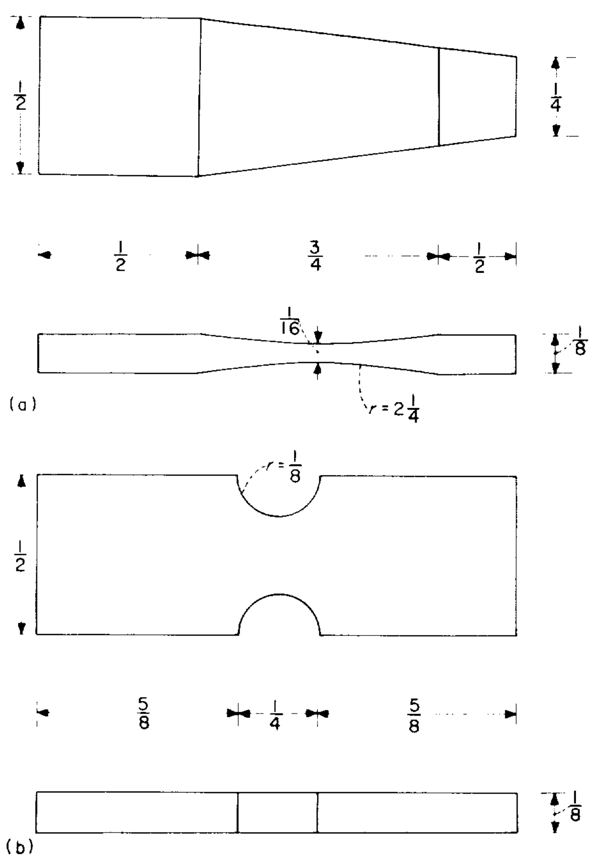
The purpose of the present investigation is to extend the work on the effects of cyclic loading on single crystals in which cross slip does not readily occur and to study the behavior of dislocations in greater detail. Single crystals of lithium fluoride (LiF) were chosen for this investigation largely because the etching techniques developed by Gilman and Johnston for this material (ref. 21) provide a means for the direct observation of dislocation behavior.

Another aim of the investigation is to study the effects of rate of cycling and type of loading on LiF specimens. The previously reported work had been done only at 1,800 cpm in fully reversed bending.

For the orientation and type of loading previously investigated it was found that the extrusion-intrusion process did not operate in LiF, and fatigue failures did not occur at stress levels an order of magnitude above the initial yield stress. This behavior has been attributed to the inability of the material to cross slip. However, this lack of ability to cross slip could serve to promote crack formation at pileups of edge dislocations, since local stress concentrations would not be relieved. In the previous work the slip planes which operated were of a nature which did not particularly favor the pileup of edge dislocations. A final aim is, therefore, to determine whether fatigue failures in suitably oriented and loaded specimens could be stress-nucleated by the pileup of edge dislocations at barriers.

SPECIMENS

The LiF crystals used in this investigation were obtained from the Harshaw Chemical Company of Cleveland as blanks cleaved or sawed to the desired orientation. Specimens were tested in bending or torsion, and the configurations that were found to be suitable, together with approximate dimensions of the crystals, are shown in figure 1. These shapes were selected because they confined the region of interest to a small area and



(a) Bending fatigue specimen.
(b) Torsion fatigue specimen.

FIGURE 1.—Specimen configurations. All dimensions are in inches.

minimized the possibility of failures occurring in the grips.

It was found that hand grinding with metallurgical papers was the most satisfactory method of shaping the crystals. Final polishing prior to annealing was done with cloths impregnated with 600 Alundum. Specimens were annealed at 1,200° F for 3 hours, followed by a furnace cool to room temperature. The specimens were checked in a polarizing microscope and found to be stress free after this anneal. The crystals were then chemically polished in a solution of ammonium hydroxide and stored in a desiccator until tested.

TESTS

BENDING TESTS

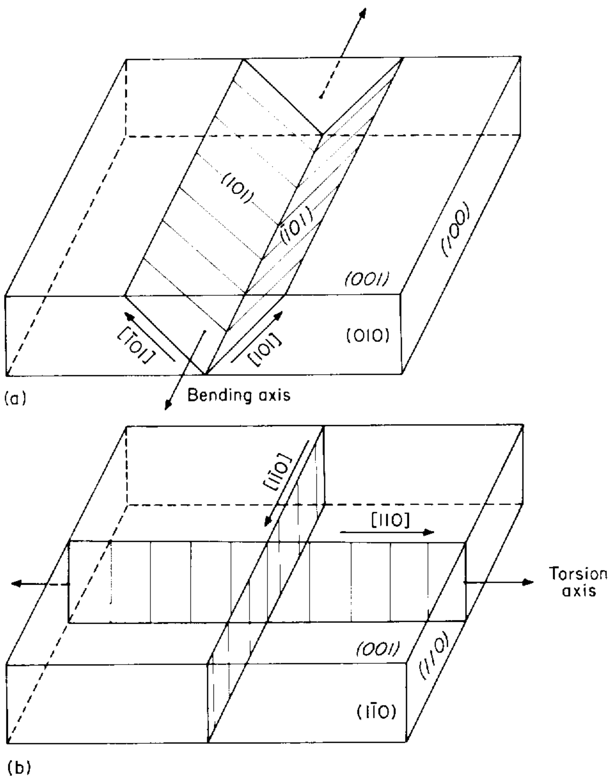
In order to check on speed effects, bending tests were run at 2 cpm and 1,800 cpm. Those at 2 cpm were constant-amplitude tests, whereas those at 1,800 cpm were constant-load tests. Fully reversed bending tests and reversed bending tests about an applied initial load or deflection were made in order to study the influence of a mean load or mean strain on the behavior of the crystals.

Specimens were tested as cantilever beams in a cantilever bending fatigue machine. The 2-cpm tests were made by utilizing the calibrated spring and dial system which are ordinarily used for applying mean loads. Deflection of the free end of the cantilever was measured by using a 30-power microscope. Stroboscopic illumination was used in the 1,800-cpm tests.

The orientation of the crystals is shown in figure 2(a) and will be described as (001) [100]. The principal slip systems are also shown in this figure. It should be remarked that two other highly stressed potential slip systems will be less likely to operate than those shown in figure 2(a) because the crystal dimension in the slip direction is much larger in the former than in the latter (ref. 22).

TORSION TESTS

The torsion tests were undertaken to determine whether edge dislocation pileups could lead to crack formation in LiF, and also to observe the behavior of dislocations by using an etching technique. The orientation of the crystals is shown in figure 2(b) and will be described as (001) [110]. The slip system for LiF is $\{1\bar{1}0\}$ $<110>$. Consequently, the slip direction lies in the face of the



(a) Bending specimen.
(b) Torsion specimen.

FIGURE 2.—Orientations of LiF specimens, with principal slip planes and slip directions indicated.

crystal in these experiments, and the operative slip systems are such that the edge dislocation portions of the loops intersect at the surface.

In these torsion tests the point of maximum shear stress falls in the surface at midwidth of the specimen, thereby eliminating edge effects and facilitating observation of the processes involved. For the configuration shown, the maximum shear stress τ_{max} is given by

$$\tau_{max}=\frac{4T}{bt^2}$$

where T is torque, b is width of specimen, and t is thickness of specimen.

Torsion testing was done in the same machine as the bending experiments with an adapter to convert to torsion loading. In general the speed of testing was 1,800 cpm, but where the number of cycles between observations was small, the torque was applied manually at about 2 cpm. (In these tests the stress level was increased slightly after every 10^5 cycles, and the amplitude of twist was measured at each stress level.)

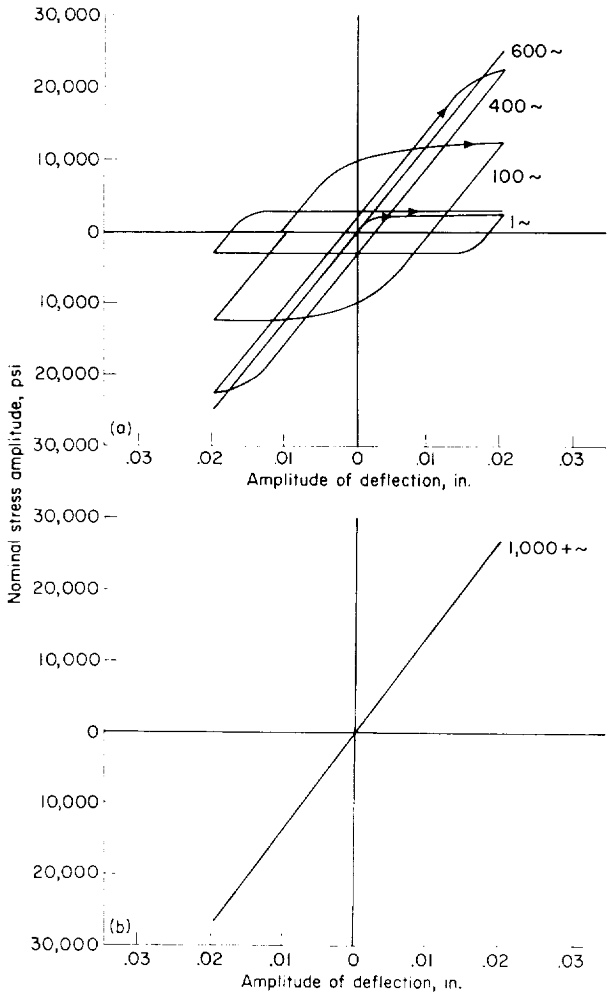
In the etch-pit studies, the procedure followed was to interrupt the test at intervals and to inspect the specimen for cracks. If none were found, the specimen was removed from the testing machine and etched in Gilman and Johnston's etch "W" (ferric fluoride in water, 1.5×10^{-4} molar) for approximately 1 minute, and then rinsed in alcohol and dried. The specimen was then photomicrographed and retested for an additional number of cycles. When cracks were found, photomicrographs were also taken prior to etching.

RESULTS

BENDING TESTS

Fully reversed bending tests.—A typical example of the results of fully reversed bending at 2 cpm is shown in figure 3(a). It is seen that after approximately 600 cycles the stress has increased from 2,500 to 25,000 psi. Bending-test results at 1,800 cpm are shown in figure 3(b). Fractures generally occurred either as a result of fretting in the grips or else upon increase of strain amplitude. In only one case did fracture occur during cycling and this case involved the largest strain amplitude, 0.085 inch. Failure of this specimen occurred after only 17 cycles. The general finding was that nothing akin to the usual type of fatigue failure occurred during these tests. Several of the specimens were etched to bring out dislocation patterns. The slipbands revealed by this technique were similar in appearance to those which will be described in greater detail in connection with the torsion tests.

Mean-load tests.—An example of the type of result obtained for tests in which the initial strain amplitude was not symmetrical about the origin is shown in figure 4. The most striking finding is that during the course of cycling the mean load is quickly eliminated. Thereafter the test is equivalent to a fully reversed bending test. The relatively low value of the initial yield stress for this material limits the amount of mean stress which could be initially applied to about 2,000 psi. However, after the yield stress of a specimen had been increased by cycling, a mean load could be sustained provided the peak stress in the cycle did not exceed the level of the new yield stress.



(a) 2 cpm.
(b) 1,800 cpm.

FIGURE 3.—Effect of cyclic speed in fully reversed bending on the behavior of LiF crystals.

Tests at 1,800 cpm yielded results similar to those obtained at 2 cpm.

REVERSED TORSION TESTS

Unetched specimens.—The primary finding of the reversed torsion cycling tests of polished but unetched specimens was that fatigue failure did not occur even though the peak shear stresses reached were of the order of 10,000 psi, as compared with an initial yield shear stress of 1,000 psi. Such failures as occurred were due to fretting in the grips or else occurred immediately upon increase of the applied torque. As in the reversed bending tests, after some cycling a linear relation

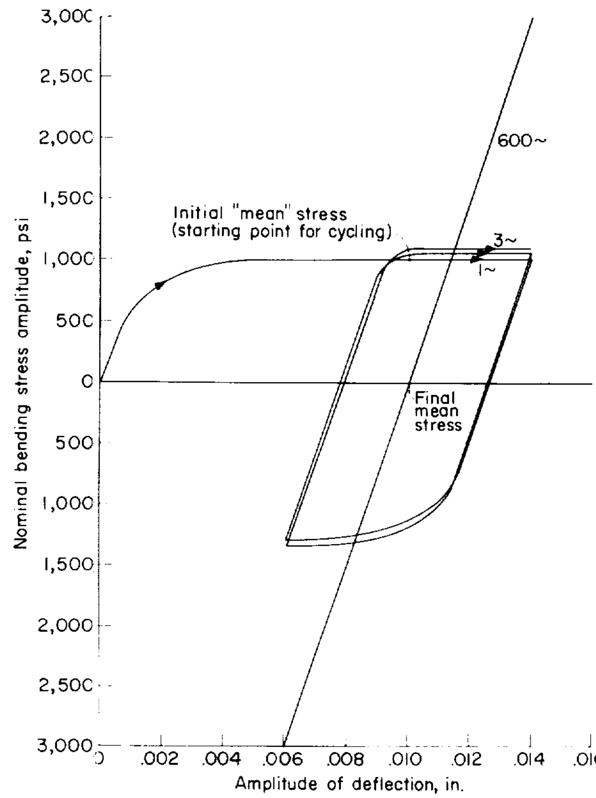
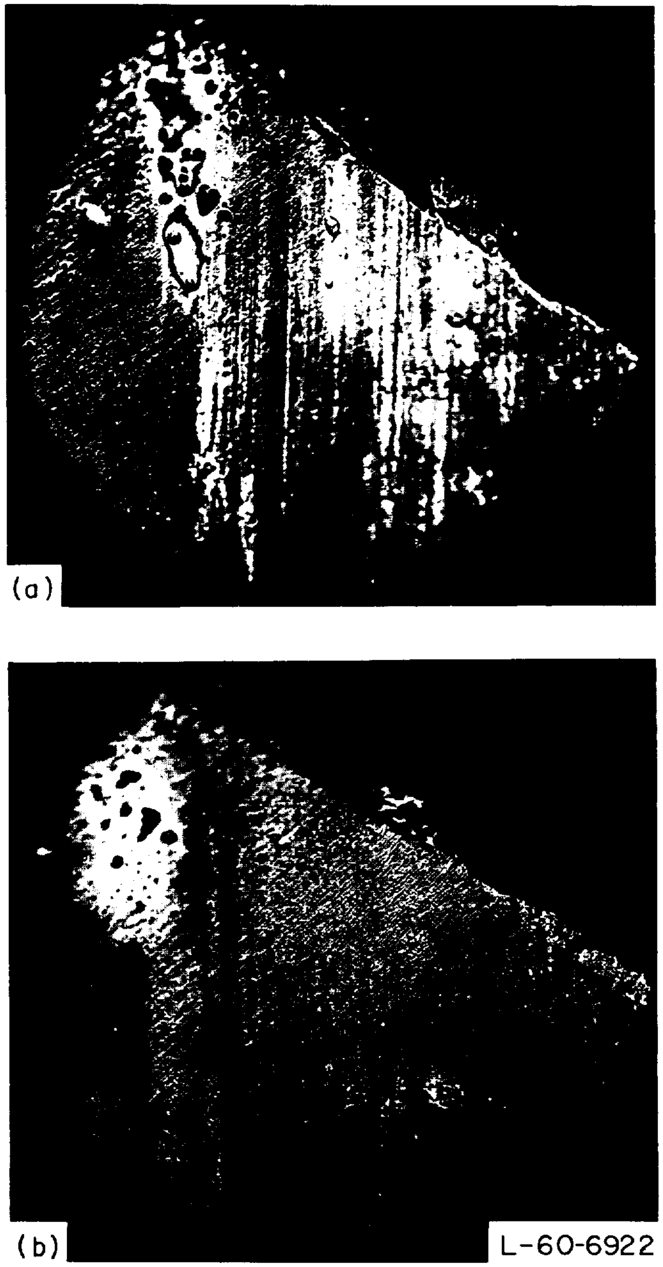


FIGURE 4.—Effect of reversed cycles of constant strain amplitude on the "mean" stress in a LiF crystal.

was found to hold between the applied torque and the amplitude of twist.

In no case was evidence of extrusions or intrusions found. However, at the higher stress levels, after some 10^7 cycles had been applied a tenacious white powder formed. An example of this effect is shown in figure 5(a), which was taken after the specimen broke. Figure 5(b) shows this same region after immersion in distilled water, and it is noted that the white powder, which, as is subsequently discussed, is believed to be lithium hydroxide has been removed and that some areas of the specimen appear to have been chemically attacked. Chemically polishing these specimens after testing revealed a persistent substructure as shown in figure 6.

Etched specimens.—Specimens etched to reveal the presence of dislocations were tested at stress levels near 1,000 psi, for at higher stress levels the density of dislocation etch pits becomes so high that interpretation is difficult. A typical example of the progressive growth of slipbands as revealed by the etch-pit technique is shown in



(a) White deposit near region of static fracture along a cubic cleavage plane.
(b) Same region as in (a) after immersion of specimen in distilled water.

FIGURE 5.—(100) surface of torsion specimen after 10^7 cycles at a shear stress of 7,500 psi. ($\times 500$)

figures 7(a) to 7(j). As seen in figure 7(a), two slip systems at right angles to each other are operative. The slipbands themselves are seen to be composed of closely spaced dislocation etch pits. Because of the orientation of the torsion specimens, the dislocations revealed by the pits are edge dislocations. Although the longitudinal and transverse bands are of similar appearance after 10 cycles, after 100 cycles it is apparent that the longitudinal bands have broadened to a greater extent than have the transverse bands.

The specimen shown in figure 7 was subjected

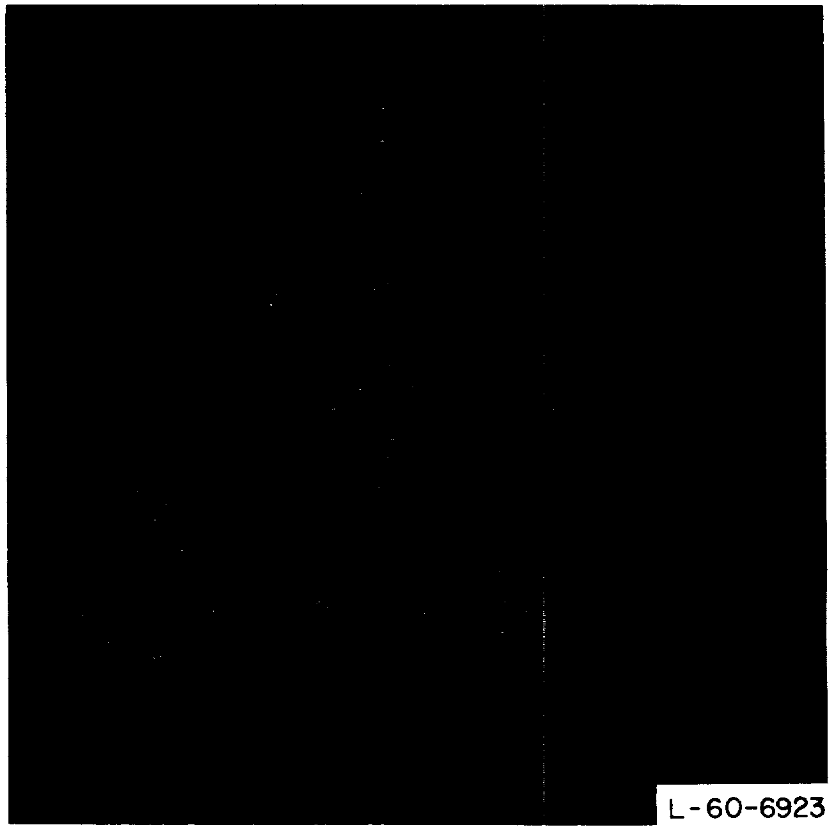


FIGURE 6.—Substructure developed beneath surface of specimen after 10^7 cycles at a shear stress of 4,000 psi. Transverse direction of specimen is parallel to straight markings. (×500)

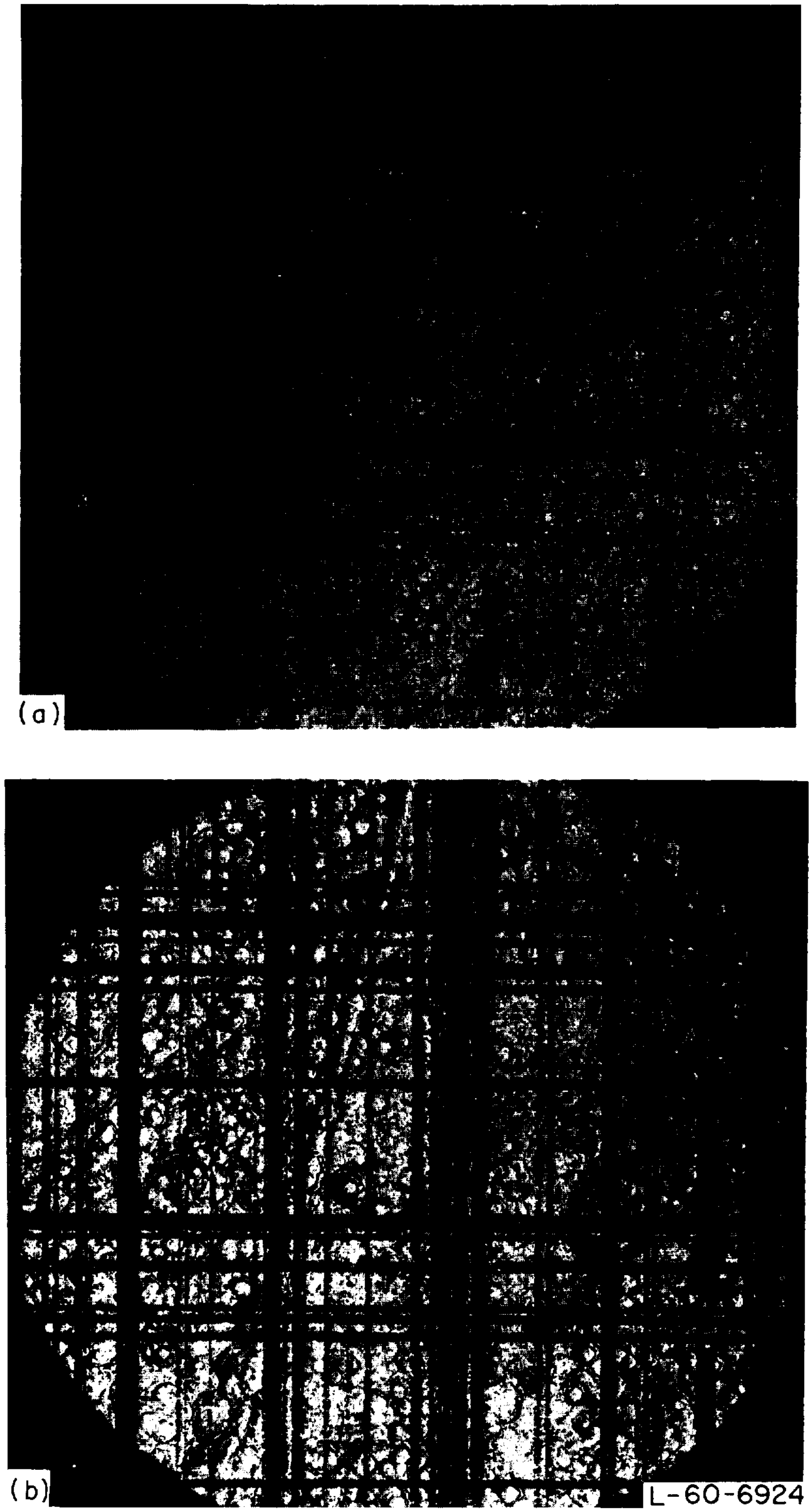
to an initial shear stress of 1,000 psi. After 1,750,000 cycles it appeared that no further changes were occurring and that cracks had not formed. The number of longitudinal slipbands was larger than after 10 cycles, although the number of transverse slipbands was unchanged. The shear stress was then increased to 1,500 psi. No cracks were detected after 100 cycles at this stress level; however, cracks were detected after 1,000 cycles. Figures 7(f) and 7(g) show the cracked region, figure 7(f) being printed so as to bring out the cracks and 7(g) so as to bring out some markings which appeared.

Figure 7(h) shows the cracked region after etching, and it is seen that severe chemical attack has occurred. This type of attack had not occurred prior to the appearance of the cracks. Chemical polishing brought out the cracks more clearly, as shown in figure 7(i). Further polishing removed the cracks completely. After the specimen was tested for an additional 10^5 cycles and then etched, additional slip lines at an angle of 45° to the original slipbands were in evidence in the cracked region, as shown in figure 7(j).

Features of interest with respect to the cracked region are as follows:

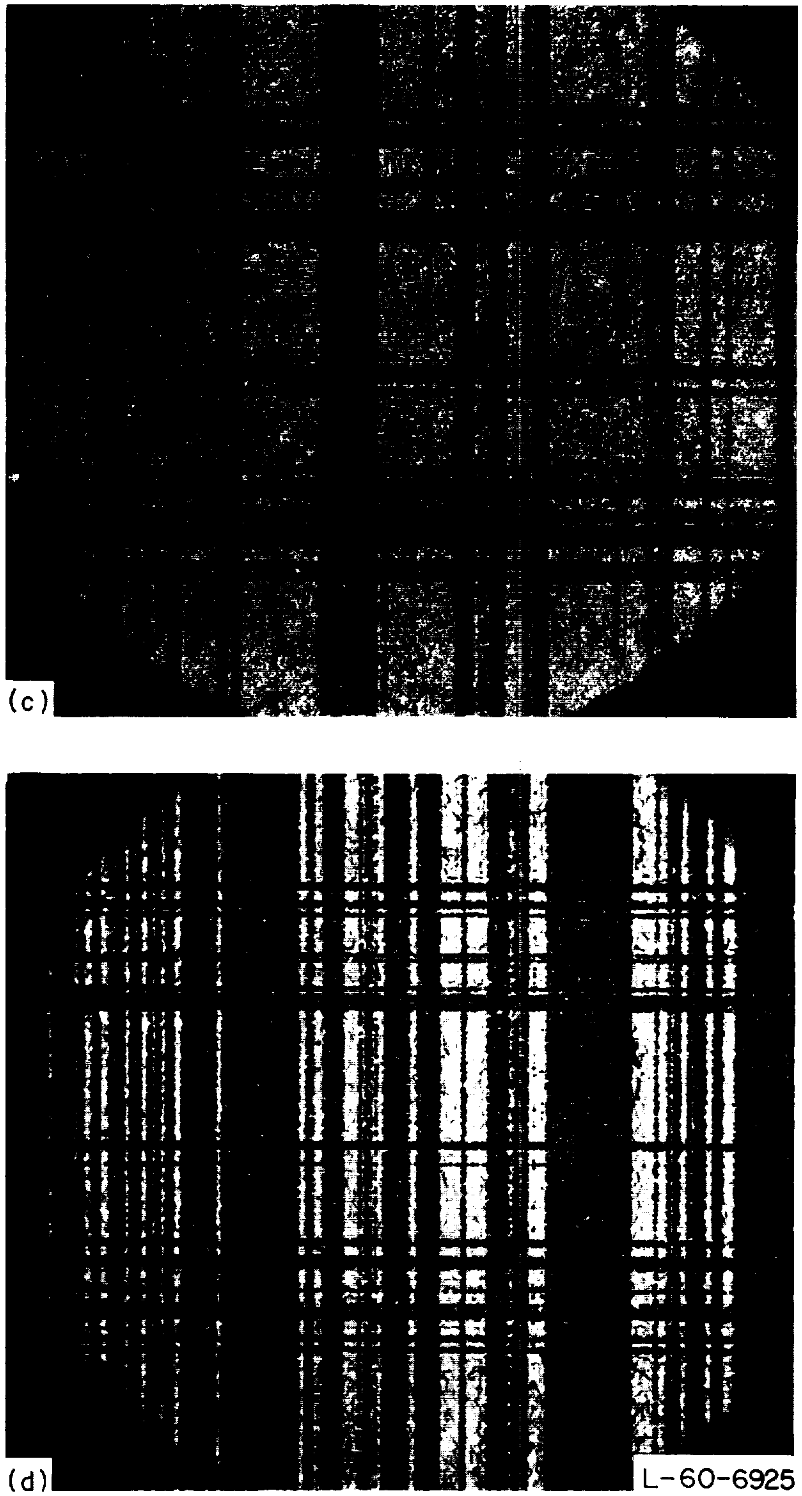
1. The cracks are perpendicular to the bands and away from the main intersections, and appear to have originated at the interface between the slipped and unslipped material. In a given band, the cracks are on the same side of the band, and while most of the cracks are in the longitudinal bands, a few are in the transverse bands. These effects may be seen best in figure 7(i), in which the light areas correspond to slipbands.

2. Simultaneously with the appearance of cracks, a change in the nature of the surface took place; in certain regions of the band etch pits are not in evidence and "slip" markings at angles of 60° to 70° to the transverse direction have appeared as indicated in figure 7(g). Immersion in the etching solution resulted in a chemical attack of the cracked region, not only in the bands but also in the unslipped regions between the bands.



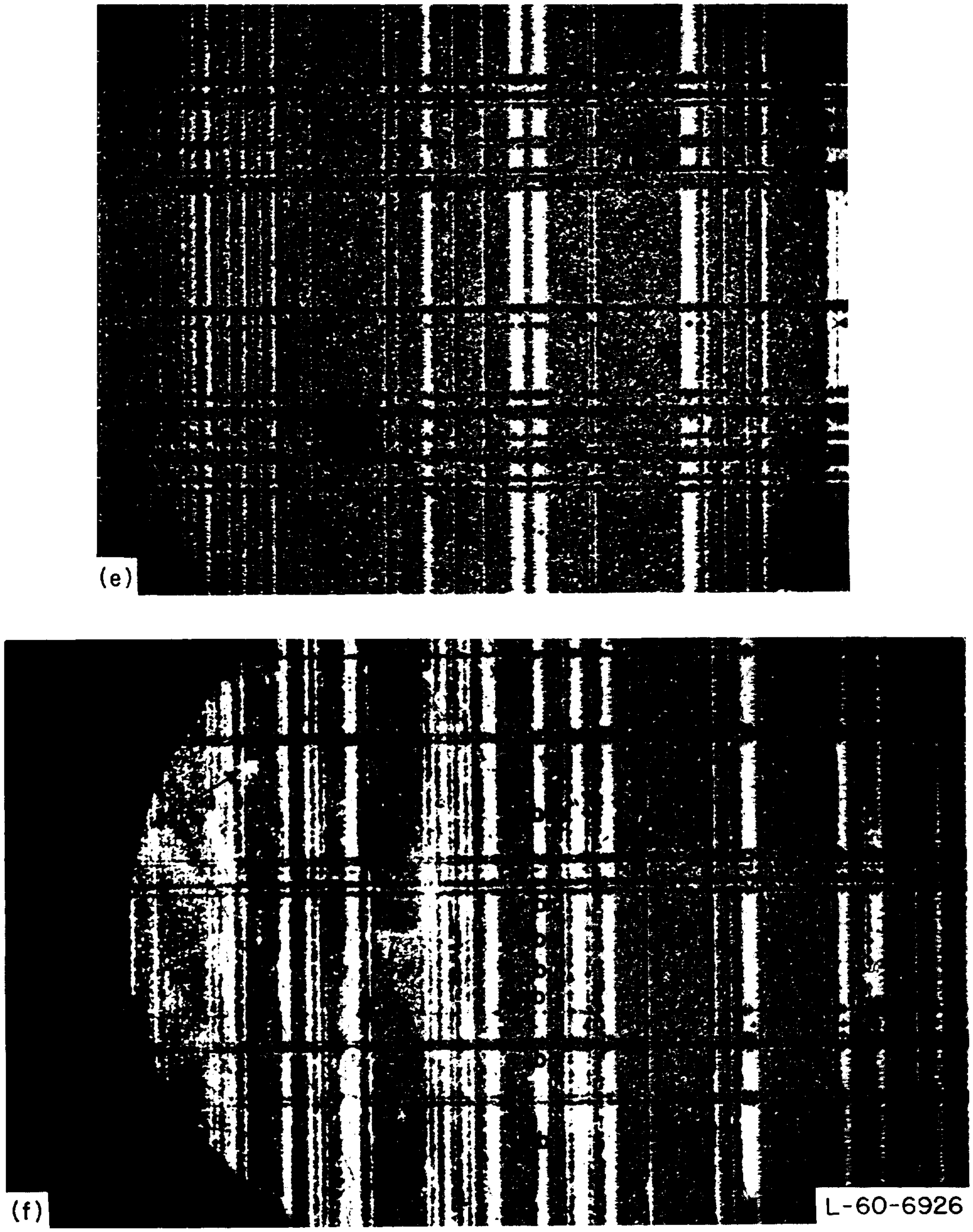
(a) After 10 cycles at a shear stress of 1,000 psi.
(b) After 10^2 cycles at a shear stress of 1,000 psi.

FIGURE 7.—Development of slipbands during torsion. Bands run in $\langle 110 \rangle$ directions. Transverse direction of crystal is horizontal. ($\times 100$)



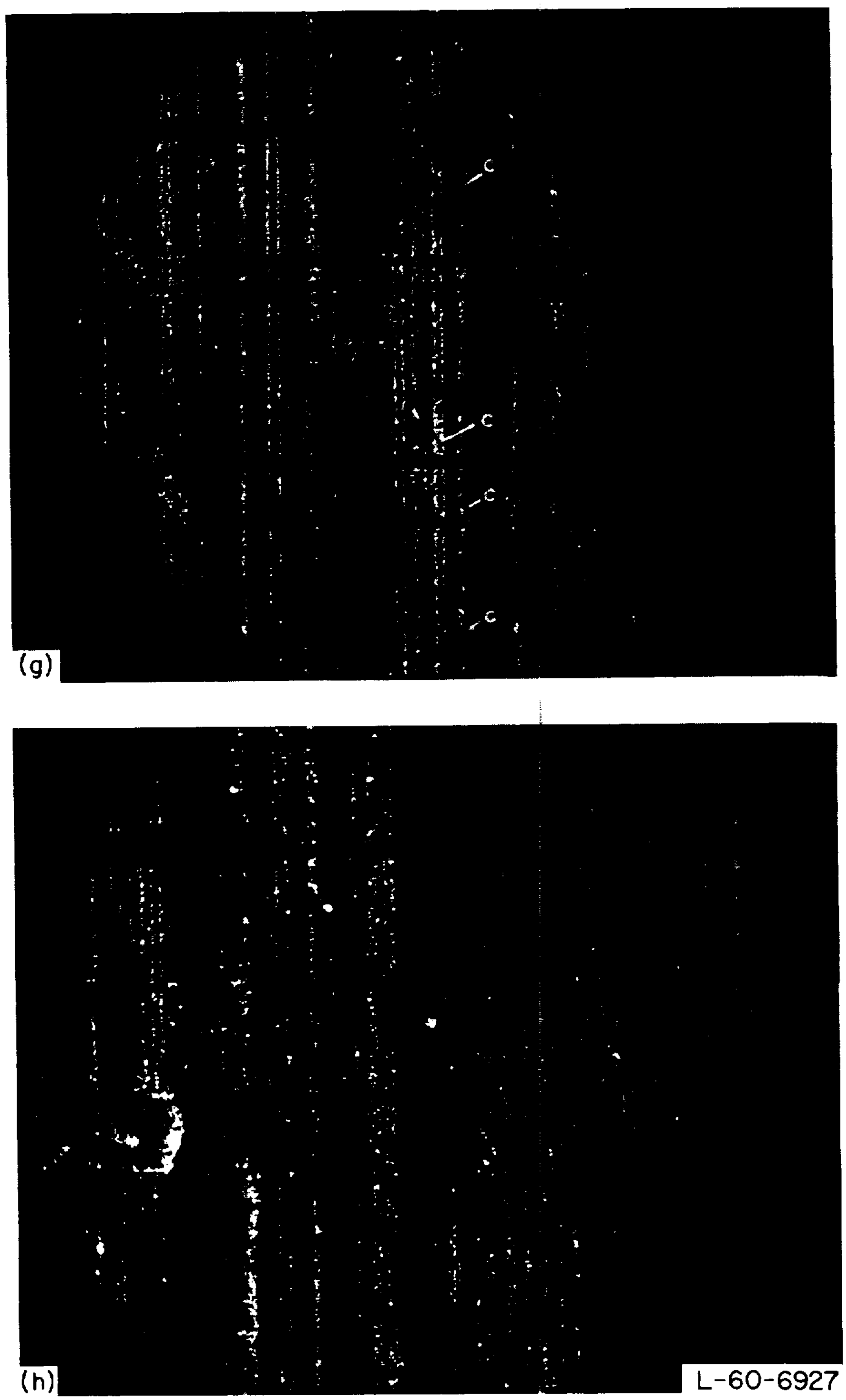
(c) After 10^3 cycles at a shear stress of 1,000 psi.
(d) After 1.25×10^6 cycles at a shear stress of 100 psi.

FIGURE 7.—Continued.



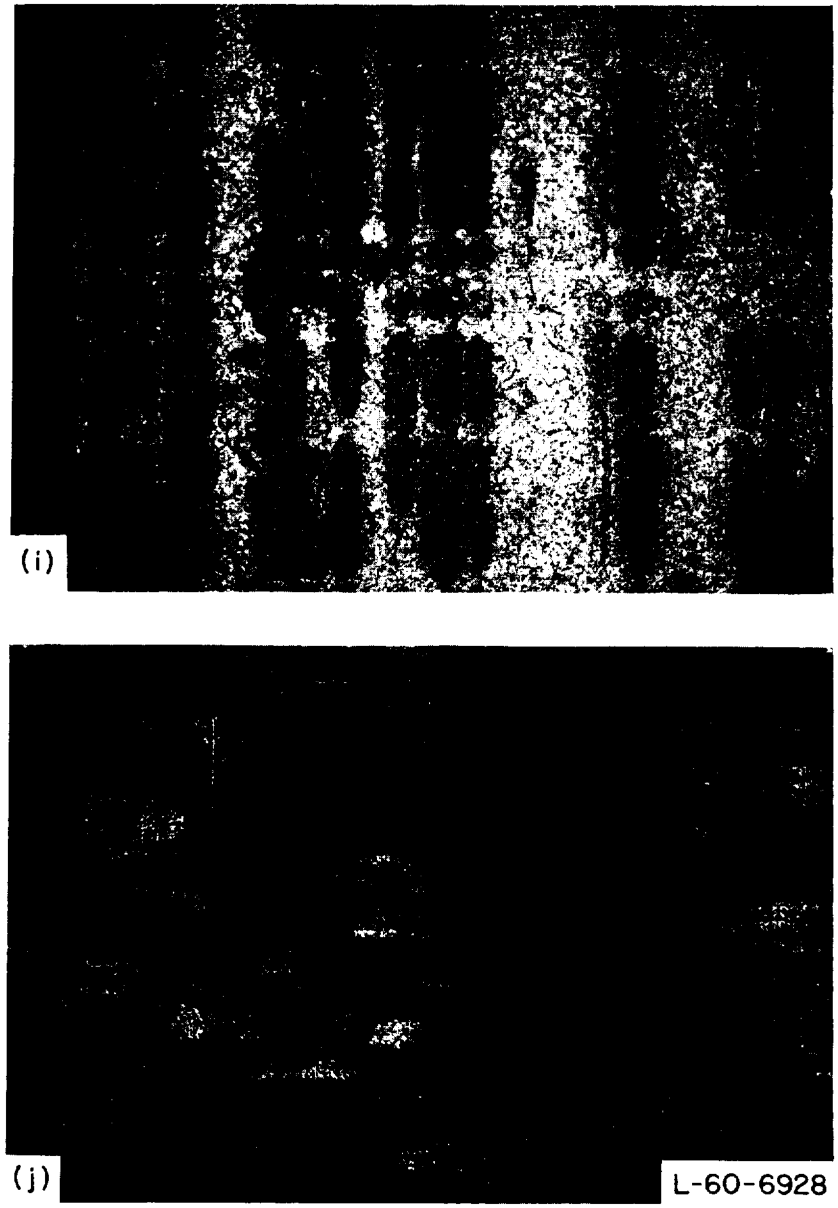
(e) After 1 additional cycle at a shear stress of 1,500 psi.
(f) After 10^3 additional cycles at a shear stress of 1,500 psi. Specimen was etched after 10^2 cycles, but not after 10^3 cycles. Typical white patches are seen at a and typical cracks at b.

FIGURE 7. Continued.



(g) Same as (f); printed to bring out 60° to 70° lines. Typical lines are indicated by the letter c.
(h) Cracked region after immersion in etching solution.

FIGURE 7.—Continued



(i) Cracked region after chemical polishing.
(j) Appearance of cracked region after removal of cracks by chemical polishing and further cycling.

FIGURE 7. Concluded.

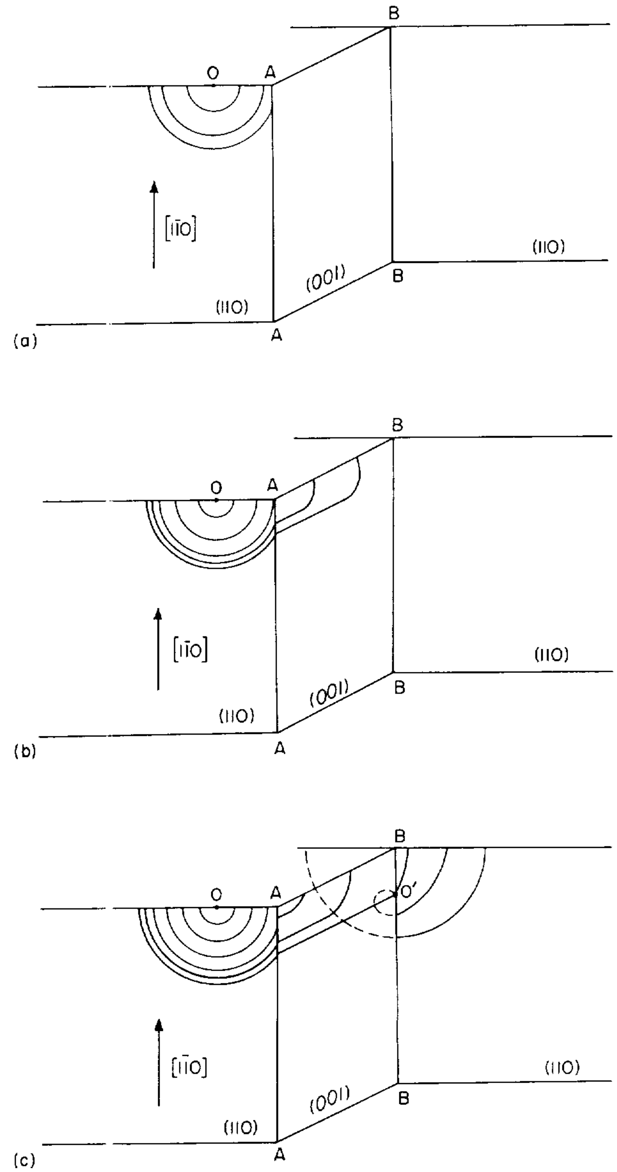
DISCUSSION OF RESULTS

BENDING

The results of the reversed bending tests at 2 cpm indicate that the cycling speed has minor influence on the behavior of LiF crystals, since the results are not significantly different from those previously obtained at 1,800 cpm (ref. 14). In both cases no fatigue failures occurred, even though there was such a marked degree of hardening that the yield strength of the crystal was raised by an order of magnitude. This increase in yield strength is definitely associated with the cycling process, for in unidirectional bending to failure very little strain hardening occurs.

The specimens were examined after testing for indications of extrusions or intrusions; however, none were found. This was to be expected in view of the inability of crystals with the sodium chloride structure to cross slip in an easy manner, since there are no two slip planes which have a common slip direction. The process of cross slip not only requires that the two slip planes involved have a common slip direction so that a screw dislocation can move from one plane to the other, but also requires that the resultant dislocation loops be free to expand in the cross-slip plane. This means that the mobility of the edge component of the expanding loop as well as the mobility of the screw component must be considered. Since the dislocations in ionic crystals such as LiF are not extended, the motion of screw dislocations out of the {110} type slip planes is expected. In fact, Gilman and Johnston (ref. 21) have obtained evidence for the motion of screw dislocations onto {100} type planes. However, Gilman (ref. 23) has also shown that glide on other than {110} planes in LiF is a very difficult process at room temperature. This must be due to the inability of edge dislocations to move on other than {110} planes.

The fact that screw dislocations are relatively free to move on other than {110} planes while edge dislocations are not is thought to account for the type of broadening of slipbands encountered in the reversed bending and reversed torsion experiments. For example, consider the situation shown in figure 8, where screw components move along a (110) plane in an LiF type of crystal until they encounter an obstacle. They then can move onto some other plane, aided in this crossing by the piling up of dislocations at the obstacle. However, the resistance of the edge component to motion on the new plane is such that the screw component returns to a (110) plane parallel to the initial plane as soon as the initial obstruction is bypassed. Slip on the cross-slip plane is quickly halted by the pileup of edge dislocations in this plane. The ends of the edge component act as Frank-Read sources, generating additional dislocations in the (110) planes. The large number of point-defect clusters treated during cyclic loading (ref. 24) may serve as barriers and lead to rapid broadening of the slipbands, until in the case of LiF at higher stress levels the entire surfaec is covered with fine slip lines. There is no circulatory motion of screw dislocations in such a material, and hence extrusions or intrusions of the type described by Mott are not expected. However, in other materials in which edge dislocations are mobile on the cross-slip plane the Mott mechanism would be expected to operate.



(a) Dislocation loops blocked at A–A by barrier.
(b) Dislocation loops cross slip on (001) where edge components are relatively immobile.
(c) Leading dislocation moves back on (110) plane. Pinning point at 0′ acts as Frank-Read source.

FIGURE 8.—Model of slipband broadening in LiF.

The tests to determine the influence of a mean load are of interest in that they show that plastic deformation will tend to eliminate the mean load, thus converting such tests to fully reversed stress tests. Since plastic deformation at critical sites is considered to be required for fatigue failure, there will be a reduction of mean stress in such critical sites. This reduction of mean stress may be the explanation for the fact that the endurance limit depends more upon range of stress than upon the nominal peak stress in the cycle.

TORSION

Unetched crystals.—The fact that fatigue failures did not occur in unetched specimens indicates that dislocation pileups do not lead to crack formation during cyclic loading of LiF specimens. (The etch-pit studies indicate that pileups do occur.) In these tests the slip vector was in the plane of the surface, but this is not considered to be responsible for the lack of fatigue failures, for in reversed bending tests the slip vector was out of the plane of the surface, but even in these tests fatigue failures did not occur. It is considered that normal fatigue failure requires that the slip vector be out of the plane of the surface, but in addition cross slip must occur.

Etched crystals.—The fact that cracking was observed only in the etched crystals indicates that cracking was facilitated by the action of the etch pits as stress raisers. The location of cracks transverse to the slipbands at the interface between slipped and unslipped material is contrary to expectations based on usual fatigue behavior. In the usual case, the cracks develop within and parallel to the bands. On the basis of the theory concerning the pileup of dislocations (refs. 25 and 26), it might be expected that cracks should initiate at the intersection of the slipbands because, as shown in figure 9, the bands themselves are barriers to slip. (Fig. 9, which shows a region of low stress in a specimen tested at 4,000 psi, also indicates that at higher stress levels the entire surface is covered by fine slip traces.) The fact

FIGURE 9.—An illustration of the barrier to dislocation motion provided by intersecting slip systems. (×100)

that cracks form away from the main intersections may indicate that some other mechanism of crack formation operated in this case.

A possibility is that cracks were initiated by the superposition of tensile stress fields of passing edge dislocations of opposite sign which were forced past each other by the increase in the stress from 1,000 to 1,500 psi. However, if this were the case, the resultant cracks should be parallel rather than perpendicular to the bands.

Another possibility is that in certain regions of the bands there is an excess of edge dislocations of one sign. Such an excess would result in a curvature of the bands, if they were free to deform. However, the surrounding unslipped material would prevent the bands from taking on this curvature, and this restraint would set up a bending stress within the band, which would act in the longitudinal direction of the band and would be a maximum at the interface between the band and the restraining unslipped material. At the lower stress level (1,000 psi), this stress coupled with the stress-raising effect of the etch pits themselves was not sufficient to cause fracture, but upon increase of stress to 1,500 psi, the density of dislocations may have been increased with a resultant increase of bending stress sufficient to initiate cracks of the type observed in these experiments.

Although it is true that there should be no excess of dislocations of one sign when averaged over the bands, it is possible that in localized regions on either side of a source, such an excess could develop. Indeed, work by Nye (ref. 27) with silver chloride indicated that an excess of dislocations of one sign was present within the bands. Recently Kear and Pratt (ref. 28) described a procedure which made use of the color changes which occurred in stressed ionic crystals when viewed under polarized light to identify the sign of the dislocations. Application of this technique to the study of slipbands developed during fatigue should prove worthwhile in determining the character of the dislocations.

Another possible explanation of the cause of crack formation is that dislocations moving along the observed new bands which make approximately 65° angles with the transverse direction are piled up at the old bands. This pileup would set up a high tensile stress which, when coupled with the already present bending stress within the bands and the stress-raising effect of the pits, could bring about the observed cracking at the edge of the original bands. Inspection of figure 7(g) reveals that many of the cracks are located at the point of intersection of the old bands with the 65° lines. In addition, the whitish regions in the slipbands, as, for example, the region located at the left center of figure 7(g), also seem to run in a 65° direction. As will be discussed in more detail subsequently, these white regions may represent excess lithium atoms corresponding to lithium vacancies created where the new slip system intersects the old, the new system having been activated upon increase of stress. Of the foregoing possibilities, it appears that this explanation is most consistent with the observations.

In addition to the development of cracks, the chemical nature of the surface of the etched specimens appeared to have been altered. Similar changes were also found in the absence of fatigue cracks on the surface of the unetched specimens both in torsion and in bending. Such specimens were chemically attacked by distilled water, the ferric fluoride etching solution, and alcohol. Since no such attack was ever found with unstressed crystals, or with unidirectionally stressed crystals it must have been associated with changes which occurred during cycling. Inasmuch as the solubility of lithium fluoride in water is very low, and lithium is soluble and has a white color, the white deposit has been tentatively identified as lithium or, more probably, lithium hydroxide, which might explain the altered appearance of the bands in figure 7(f) as due to the combination of lithium with environmental moisture to form lithium hydroxide. If the deposit is indeed lithium hydroxide, then the production of excess lithium at the surface must involve the simultaneous production of lithium vacancies or fluorine interstitials. On the basis of the work of Lehovec (ref. 29) and of Pratt (ref. 30), the energy required to form a lithium vacancy is less than that required to form a fluorine interstitial; hence vacancy formation would be more likely to occur. The experimental work on unetched specimens indicates that large numbers of vacancies were formed without the formation of fatigue cracks, and this finding provides a further basis for rejection of the speculation that vacancies are responsible for rapid fatigue failure.

In the tests involving etched specimens, the greater broadening of the longitudinal bands can be attributed to the greater dimension of the longitudinal direction in comparison with that of the transverse direction. As discussed in the previous section, slipband broadening is considered to require cross slip where slip in the primary system is blocked by an obstacle. In crystals of LiF, however, this cross slip is of a very limited nature because of the immobility of the edge component of an expanding dislocation loop in the cross-slip plane. If the number of obstacles to primary slip is proportional to the length involved, then it is reasonable to expect the greater broadening of the longitudinal bands. This explanation would not apply, however, to the observation that the number of longitudinal bands also increased, whereas the number of transverse bands did not.

CONCLUDING REMARKS

In reversed bending and reversed torsion tests on a number of single crystals of lithium fluoride the principal findings were as follows:

With reversed stress in bending:

1. There was no significant difference between the behavior of specimens tested at 2 cpm and 1,800 cpm.
2. The strength of the LiF crystals was increased by an order of magnitude as a result of cycling, and no fatigue failures were obtained.
3. Plastic deformation during the course of cycling tends to eliminate any mean loads which may have been initially imposed.

With reversed stress in torsion:

1. In polished but unetched LiF specimens no fatigue failures occurred and no extrusions or intrusions were found.
2. Cracks were found only in those crystals that contained dislocation etch pits.
3. The amplitude of deformation was directly proportional to the amplitude of applied torque up to the maximum stress applied.
4. The maximum stress applied was an order of magnitude higher than the static yield stress of the virgin crystal.
5. Evidence of vacancy formation was found, but the presence of vacancies did not result in crack formation.

It is concluded that:

1. The present results furnish additional evidence that easy cross slip is required for fatigue failure to occur in its normal fashion.
2. A variation in speed of cycling over the range from 2 cpm to 1,800 cpm has no significant effect on the behavior of LiF.
3. Vacancy formation did not lead to fatigue failure in these experiments.
4. Plastic deformation serves to eliminate mean loads.
5. Dislocation pileups did not lead to the formation of fatigue cracks in these experiments.

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